

Removal and Recovery of Nutrients as Struvite from Anaerobically Digested Slaughterhouse Wastewater

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ABSTRACT

This study investigated the removal and recovery of ammonium (NH_4^+) and phosphate (PO_4^{3-}) from anaerobically digested slaughterhouse wastewater as struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$). Nutrients removal experiments were conducted by the addition of magnesium sulfate MgSO_4 as a Mg^{2+} source to the wastewater. The effects of $\text{Mg}^{2+} : \text{PO}_4^{3-}$; $\text{Mg}^{2+} : \text{NH}_4^+$; $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ molar ratios and pH on NH_4^+ and PO_4^{3-} removal were monitored. To verify the recovery of NH_4^+ and PO_4^{3-} as struvite, the precipitates obtained during the experiments were analyzed by X-ray diffraction. The results showed that the increase of Mg^{2+} concentration and raising the pH to 10 leads to a significant improvement of NH_4^+ and PO_4^{3-} removal. The molar ratio of $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-} = 1 : 19 : 0.58$ and a pH=9 gave optimal removal of NH_4^+ and PO_4^{3-} . The characterization of the precipitates by X-ray diffraction showed the presence of struvite crystals, confirming the recovery of NH_4^+ and PO_4^{3-} in struvite.

Key words: Nutrient removal, Nutrient recovery, Struvite precipitation, Slaughterhouse wastewater.

1. INTRODUCTION

Ammonia and phosphate are harmful pollutants for the aquatic environment. The discharge of these pollutants into surface waters is responsible for eutrophication, which leads to a depletion of oxygen in the aquatic environment. In light of this, the removal of ammonium and phosphate from wastewater has received considerable attention from researchers [1]. Furthermore, the depletion of natural phosphate reserves and the increasing need for nitrogen, phosphorus, and potassium for intensive agriculture make ammonium and phosphate-rich wastewater a valuable nutrient source [2].

Slaughterhouse wastewater after treatment by anaerobic digestion is loaded with high levels of ammonium and phosphate [3]. For nutrient removal from wastewater, various biological and chemical processes have been developed. Biological methods include constructed wetland systems, biological processes for phosphorus removal and nitrification-denitrification methods. Although these processes are commonly employed, they do not have a nitrogen and phosphorus recovery potential. Chemicals include coagulation-flocculation and electrocoagulation, which are effective methods for phosphate removal [4,5]. However, these methods remain completely ineffective for ammonium removal [6]. In general, most of the above methods can remove only ammonium or phosphate but not both pollutants simultaneously. Thus, an alternative method for the simultaneous removal of ammonium and phosphate is necessary. To solve this problem, struvite precipitation is the best solution because it allows the simultaneous removal of ammonium and phosphate. This method

allows an efficient removal of nitrogen and phosphate from wastewater by recovering them in the form of struvite and also provides a solution to the depletion of phosphate resources.

Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is a crystalline substance consisting of Mg^{2+} , NH_4^+ and PO_4^{3-} in a molar ratio of $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-} = 1 : 1 : 1$. This is a high-quality fertilizer due to its slow nutrient release [7]. The reaction of struvite formation is as follows [8,9]: $\text{Mg}^{2+} + \text{NH}_4^+ + \text{H}_n\text{PO}_4^{n-3} + 6\text{H}_2\text{O} \rightarrow \text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O} + n\text{H}^+$ ($n = 0, 1$ ou 2) Precipitation of struvite often occurs in wastewater when ammonium, phosphate, and magnesium ions exceed the struvite solubility limit. The precipitation of struvite depends on two main factors, which are the molar ratio of $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ and pH [10,11]. Several works on the removal of NH_4^+ et de PO_4^{3-} as struvite have been performed. By studying the precipitation of struvite in a UASB reactor treating poultry manure wastewater, Yetilmezsoz and Sapci-Zengin [12] obtained a maximum NH_4^+ removal of 85.4%

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ISSN NO: 2320-0898 (p); 2320-0928 (e)
DOI: 10.22607/IJACS.2023.1102010

Received: 26th December 2022;

Revised: 24th March 2023;

Accepted: 25th March 2023.

at pH = 9.0. Yilmazel and Demirer [13] evaluated the removal of nitrogen (N) and phosphorus (P) by struvite crystallization from liquid poultry manure residues pretreated by anaerobic digestion. These showed that the increase in molar ratio improved NH_4^+ removal with an average of 86.4% for a Mg: N:P molar ratio of 1:1:1 compared to 97.4% NH_4^+ for a Mg:N:P molar ratio of 1.5:1:1. However, although numerous works [14,15] have shown that the increase of $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ molar ratios improves the removal of NH_4^+ et de PO_4^{3-} , the uncontrolled addition of large amounts of Mg^{2+} , NH_4^+ , PO_4^{3-} may result in high residual concentrations in treated wastewater [16]. For digested slaughterhouse wastewater that contains low concentrations of Mg^{2+} [3], the addition of Mg source in sufficient quantity to precipitate struvite must be performed. Thus, the main objective of this study is to optimize the removal of ammonium and phosphate in the form of struvite only by the addition of Mg^{2+} to the wastewater to be treated.

Thus, the main objective of this study is to remove ammonium and phosphate, mainly in the form of struvite by the addition of Mg^{2+} to wastewater. Struvite precipitation experiments from digested slaughterhouse wastewater will be conducted to determine the optimal treatment conditions, including $\text{Mg}^{2+} : \text{PO}_4^{3-}$; $\text{Mg}^{2+} : \text{NH}_4^+$; $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ molar ratios and pH. Then, the characterization of the formed precipitates will confirm the production of struvite. This study will allow us to propose simple and efficient solutions for the removal of ammonium and phosphate from digested slaughterhouse wastewater in the form of struvite by using moderate amounts of Mg^{2+} .

2. EXPERIMENTAL

2.1. Digested Slaughterhouse Wastewater

The digested wastewater sample was obtained from an anaerobic digestion reactor operating under mesophilic temperature conditions. During sampling, the supernatant was collected to reduce suspended solids. The digested wastewater was then filtered through a 0.45 μm Whatman filter to remove residual suspended solids, and the wastewater sample was characterized before treatment. The characteristics of the digested wastewater are presented in Table 1. The effluent had low

Table 1: Characteristics of digested slaughterhouse wastewater.

| Parameters | Values |
|------------------------------------|-----------|
| pH | 8.03±0.02 |
| Conductivity (ms/cm) | 7.92±0.04 |
| COD (mg/L) | 5974±276 |
| BOD (mg/L) | 1200±100 |
| BOD/COD ratio | 0.20±0.01 |
| PO_4^{3-} (mg/L) | 64±1.15 |
| NH_4^+ (mg/L) | 1239±39 |
| NO_3^- (mg/L) | 8.3±0.6 |
| TA (mg CaCO_3/L) | 5147±77 |
| Mg^{2+} (mg/L) | 6.07 |
| Ca^{2+} (mg/L) | 28.05 |
| VFA (mg CaCO_3/L) | 1645±31 |
| VFA/TA ratio | 0.32±0.01 |

biodegradability with a BOD/COD ratio of 0.20, and high concentrations of NH_4^+ and PO_4^{3-} against a low concentration of Mg^{2+} . Furthermore, a high alkalinity of the wastewater is observed, as well as a high load of dissolved organic matter.

2.2. Batch Experiments

2.2.1. Effect of molar $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ ratio

The objective of these tests was to determine the optimal $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ molar ratio favorable for better removal of NH_4^+ and PO_4^{3-} in the form of struvite. Thus, the effect of $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ molar ratio on the removal of NH_4^+ and PO_4^{3-} was studied. For experiments, a volume of 300 mL of wastewater was used. Due to the low initial magnesium concentrations in digested wastewater (Table 1), appropriate masses of magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) were added to the wastewater sample to obtain the molar ratios $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ as follows: (1: 60: 1.81), (1: 49: 1.47), (1: 40: 1.17), (1: 30: 0.91), (1: 19: 0.58), (1: 10: 0.30) and (1: 5: 0.15). After the addition of Mg^{2+} to the 300 mL of wastewater, the mixture was stirred at a speed of 175 rpm for 3 min and then at 175 rpm for 60 min at room temperature (30°C). The mixture was allowed to settle for 60 min and the supernatant was collected. Finally, residual concentrations of NH_4^+ , PO_4^{3-} and Mg^{2+} were measured.

2.2.2. Effect of pH

For the determination of the optimal pH favorable for better removal of NH_4^+ and PO_4^{3-} in the form of struvite, different initial pH values of the wastewater were used. The pH was adjusted to 7; 8; 9; 10 and 11 using 4M H_2SO_4 and 2 M NaOH solutions. These tests were performed using the optimal molar ratio $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ obtained. After the addition of Mg^{2+} , the mixture was stirred at 175 rpm for 3 min and then at 175 rpm for 60 min at room temperature (30°C). The mixture was then allowed to settle for 60 min and then the supernatant was collected and the residual concentrations of NH_4^+ , PO_4^{3-} and Mg^{2+} were analyzed. The evolution of the residual concentrations of NH_4^+ , PO_4^{3-} and Mg^{2+} allowed us to determine the optimal pH for the removal of NH_4^+ and PO_4^{3-} in the form of struvite. Finally, to confirm the formation of struvite crystals, the optimal $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-}$ molar ratio and pH were used for the treatment of the digested wastewater. After treatment, the precipitates produced were collected, dried at room temperature and then characterized by X-ray diffraction (XRD).

2.3. Analytical Methods

The parameters in this study were measured according to standard methods [17]. NH_4^+ was determined by the distillation-titration method. During this method, NH_4^+ titration was performed with a 0.1 N sulfuric acid (H_2SO_4) solution after distillation of 50 mL of the wastewater sample. For PO_4^{3-} measurement, the sample was previously acidified with 0.1 N H_2SO_4 solution to a pH 2-7. Then, the analysis was done by ascorbic acid method with a Shimadzu UV-1601 spectrophotometer. Ca^{2+} and Mg^{2+} were determined according to the method proposed by [17]. The precipitates produced during processing were characterized by an X-ray diffractometer (XRD Rigaku MiniFlex II). The phases of the XRD diagram were identified using the International Center for Diffraction Data (ICDD) database (PDF#71–2089). The phosphate and ammonium removal efficiencies, expressed as a percentage, were calculated by using their concentrations before and after struvite precipitation. The standard error was calculated from duplicate experiments and expressed as error bars in the relevant figures of experimental results.

3. RESULTS AND DISCUSSION

3.1. Effect of Molar $Mg^{2+} : NH_4^+ : PO_4^{3-}$ Ratio

The effect of the Mg^{2+}/PO_4^{3-} molar ratio on phosphate removal from digested slaughterhouse wastewater is shown in Figure 1. The Mg^{2+}/PO_4^{3-} molar ratio was calculated as a function of the initial concentration of PO_4^{3-} in the digested water and the added $MgSO_4$. The removal efficiency of PO_4^{3-} is improved when the concentration of Mg^{2+} increases, in other words, when the molar ratio of Mg^{2+}/PO_4^{3-} increases. The maximum removal of PO_4^{3-} is obtained with the molar ratio of $Mg^{2+}/PO_4^{3-} = 1 : 0.15$ resulting in an abatement of 52.51% as shown in Figure 2. This result is similar to that observed by [18], who

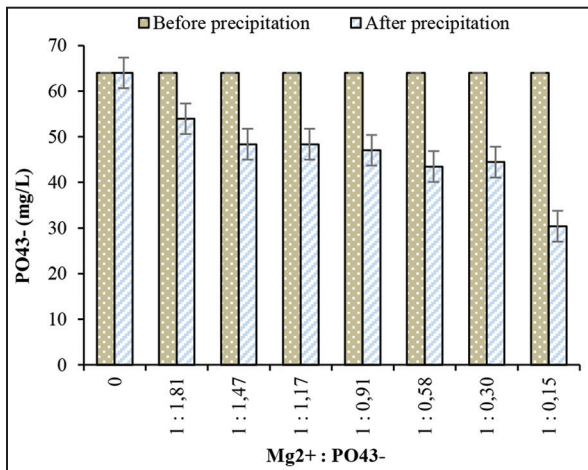


Figure 1: Evolution of the concentration of PO_4^{3-} as a function of the molar ratio $Mg^{2+} : PO_4^{3-}$.

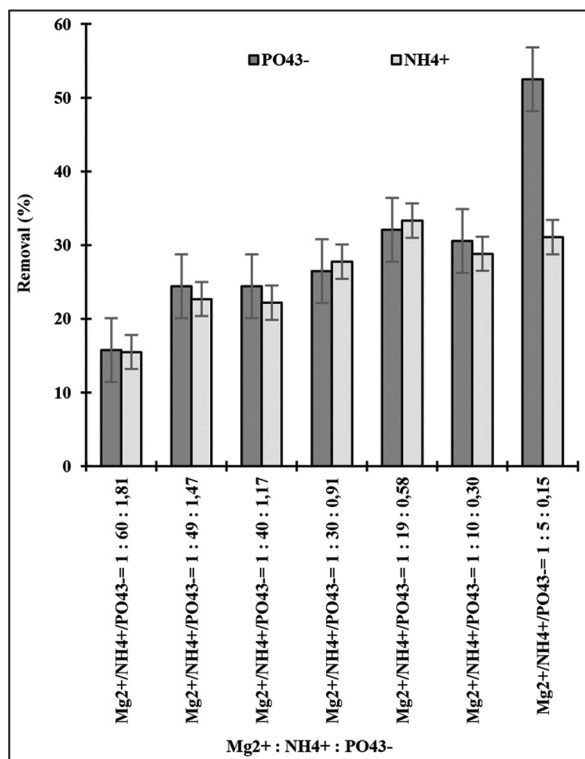


Figure 2: Abatements of PO_4^{3-} and NH_4^+ as a function of molar ratio $Mg^{2+} : NH_4^+ : PO_4^{3-}$.

obtained a removal of 55% of soluble phosphorus from swine lagoon wastewater in the form of struvite and calcium phosphates at pH 8.5. However, with the molar ratio $Mg^{2+}/PO_4^{3-} = 1 : 0.15$, the residual Mg^{2+} concentration in the treated water increases rapidly with the value of 122.26 mg/L as shown in Figure 3.

The effect of the molar ratio of Mg^{2+}/NH_4^+ on NH_4^+ removal is shown in Figure 4. As with PO_4^{3-} , the increase in Mg^{2+} concentration improves the removal of NH_4^+ . Beyond the molar ratio of $Mg^{2+}/NH_4^+ = 1 : 19$, the increase of Mg^{2+} concentration does not lead to an improvement of NH_4^+ removal. With the molar ratio of $Mg^{2+}/NH_4^+ = 1 : 19$, a removal of 33.33% is obtained (Figure 2) with a low residual Mg^{2+} concentration of 29.6 mg/L.

The analysis of Figures 1 and 4 that the increase in the molar ratio of $Mg^{2+} : NH_4^+ : PO_4^{3-}$ simultaneously improves the removal of NH_4^+ and PO_4^{3-} . This result is consistent with that of Huang *et al.* [19] obtained by studying the effect of Na^+ attenuation on phosphate and potassium recovery by crystallization of K-struvite from synthetic urine. However, for molar ratios greater than $Mg^{2+} : NH_4^+ : PO_4^{3-} = 1 : 19 : 0.58$, the increase in Mg^{2+} concentration does not result in an improvement in the simultaneous removal of NH_4^+ and PO_4^{3-} . This increase in molar ratio $Mg^{2+} : NH_4^+ : PO_4^{3-}$ is favorable only to the removal of PO_4^{3-} as shown by the molar ratio $Mg^{2+} : NH_4^+ : PO_4^{3-} = 1 : 5 : 0.15$. Furthermore, a rapid increase in residual Mg^{2+} concentrations is observed beyond the molar ratio $Mg^{2+} : NH_4^+ : PO_4^{3-} = 1 : 19 : 0.58$ (Figure 3). This result shows that beyond this molar ratio, the addition of Mg^{2+} is not necessary and may constitute a pollution of the treated water as it leads to a high residual Mg^{2+} concentration as observed in the work of Wang *et al.* [20]. The simultaneous decrease in NH_4^+ and PO_4^{3-} concentrations could be attributed to struvite formation. Recalling that the objective of this study was to remove NH_4^+ and PO_4^{3-} as struvite while minimizing the

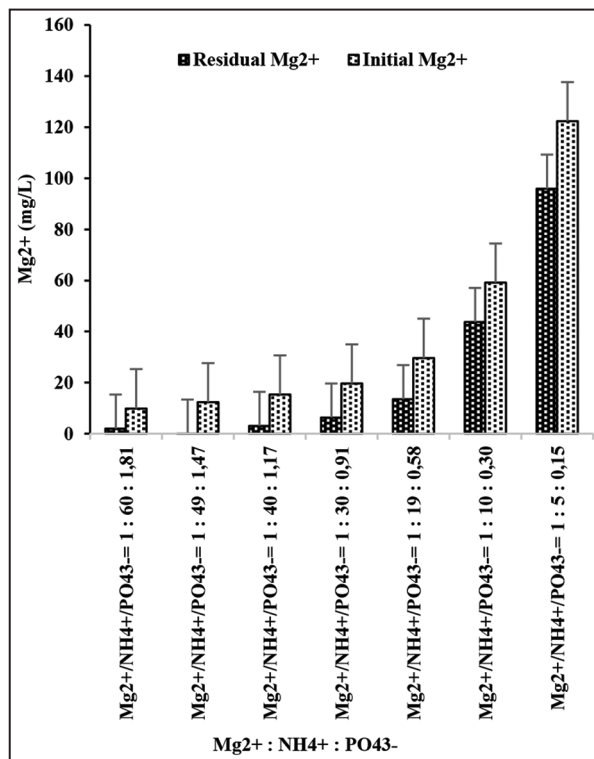


Figure 3: Initial and residual Mg^{2+} concentrations as a function of molar ratio $Mg^{2+} : NH_4^+ : PO_4^{3-}$.

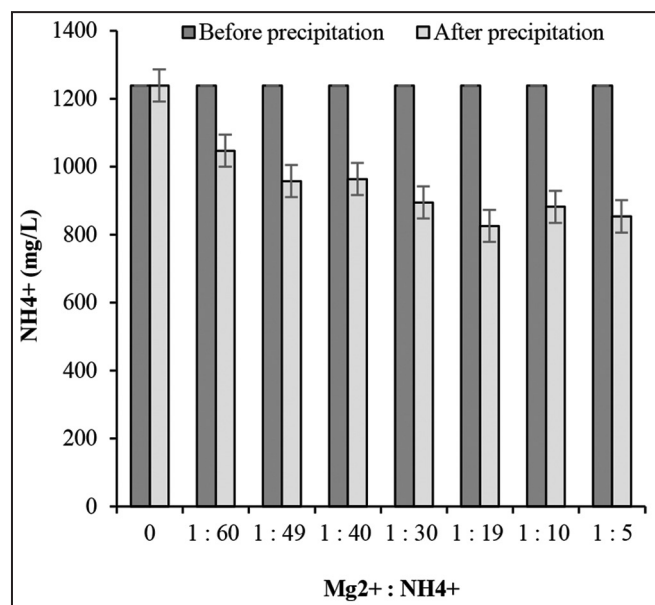


Figure 4: Evolution of the concentration of NH₄⁺ as a function of the molar ratio Mg²⁺ : NH₄⁺.

residual Mg²⁺ concentration, the molar ratio of Mg²⁺ : NH₄⁺ : PO₄³⁻ = 1: 19: 0.58 was identified as the optimal molar ratio.

3.2. Effect of pH

pH is considered a crucial parameter that affects the struvite precipitation process [9,21]. For the optimization tests, the experiments were performed with the optimal molar ratio Mg²⁺ : NH₄⁺ : PO₄³⁻ = 1: 19: 0,58. Figures 5-7 shows the evolution of Mg²⁺, NH₄⁺, PO₄³⁻ and Ca²⁺ concentration as a function of pH, the abatement of Mg²⁺, NH₄⁺, PO₄³⁻ et Ca²⁺ concentration as a function of pH, and the evolution of final pH as a function of initial pH, respectively.

As shown in Figure 6, the removal of PO₄³⁻, NH₄⁺, Mg²⁺, and Ca²⁺ is better with an increase in pH. When pH increases from 7 to 9, a simultaneous removal of Mg²⁺, NH₄⁺ and PO₄³⁻ is observed, with abatements of 41.93% NH₄⁺, 80.86% PO₄³⁻, and 36.85% Mg²⁺ at pH = 9. Except for pH = 7, the decrease in final pH is observed in Figure 7. At pH = 10, an improvement in the removal of NH₄⁺, Mg²⁺ and Ca²⁺ with 69.51, 98.63, and 52.23%, respectively, is achieved. However, the removal of PO₄³⁻ (78.26%) at pH = 10 remains identical to that at pH = 9. Finally, at pH = 11, Ca²⁺ is completely removed.

The distribution of different species of Mg, P, and N in the forms of Mg²⁺, MgOH⁺, MgPO₄⁺, H₃PO₄, H₂PO₄⁻, HPO₄²⁻, PO₄³⁻, NH₃, and NH₄⁺ in solution is determined by pH [22]. For pH 8 to 9, simultaneous removal of NH₄⁺, PO₄³⁻ and Mg²⁺ ions would result from struvite formation. According to Hu *et al.* [23], this is explained on the one hand by the presence of NH₄⁺, PO₄³⁻ and Mg²⁺, which are the essential forms for struvite precipitation. On the other hand, the drop in pH during the tests is seen as an indicator of struvite precipitation [24].

At pH = 10, the improvement in the removal of NH₄⁺, PO₄³⁻ and Mg²⁺ ions would result from several chemical reactions, including the formation of struvite, hydroxyapatite (Ca[PO₄]₃OH[s]), magnesium hydroxide Mg(OH)₂ and the conversion of NH₄⁺ to NH₃. Indeed, the work of Zyman *et al.* [25] showed that at pH >9, calcium phosphates are converted to hydroxyapatite. In addition, NH₄⁺ are converted to

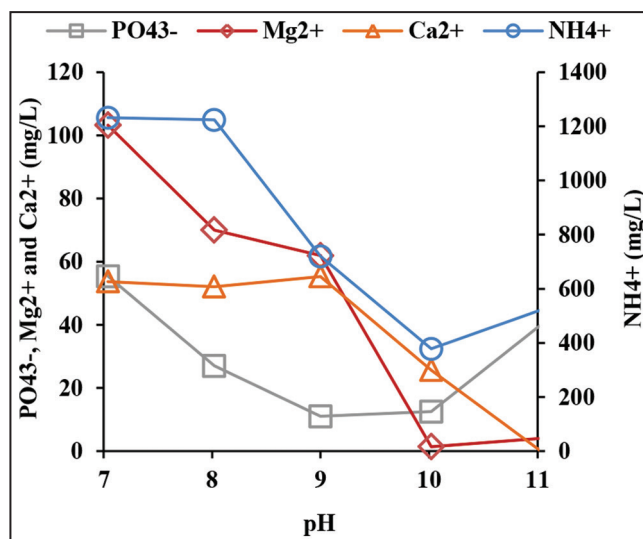


Figure 5: Evolution of Mg²⁺ : NH₄⁺ : PO₄³⁻ and Ca²⁺ concentrations as a function of pH.

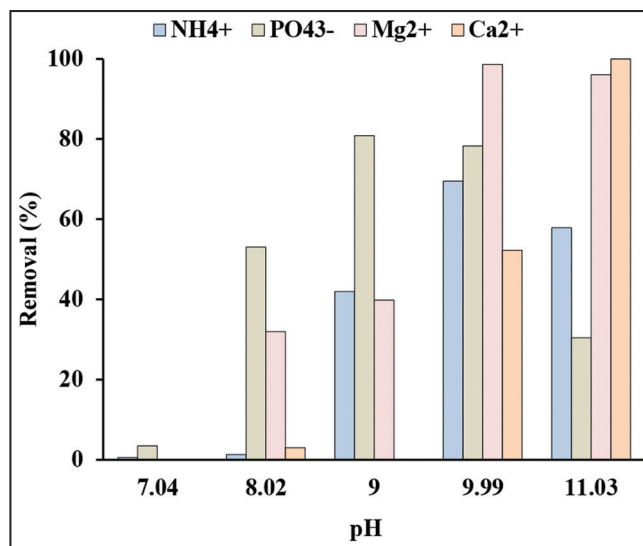


Figure 6: Abatement of Mg²⁺, NH₄⁺, PO₄³⁻ and Ca²⁺ as a function of pH.

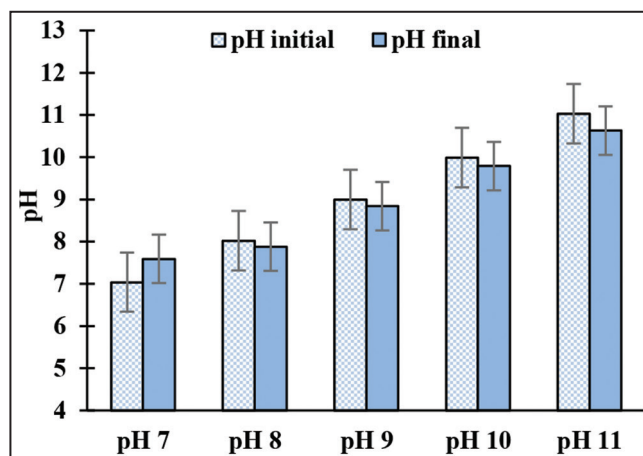


Figure 7: Evolution of the final pH as a function of the initial pH.

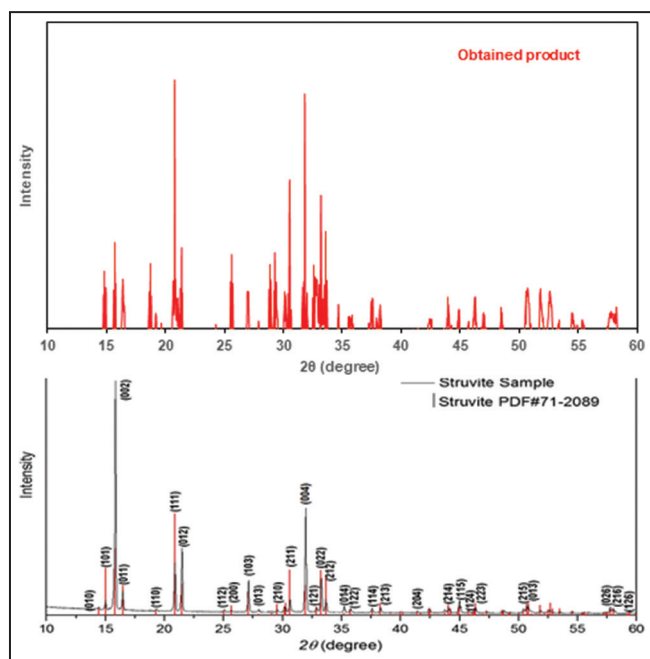


Figure 8: Diffraction of the obtained product.

NH_3 [26], resulting in a decrease in NH_4^+ and PO_4^{3-} removals as struvite [23]. At pH = 11, the removals of NH_4^+ , PO_4^{3-} and Mg^{2+} in the form of struvite are reduced in benefit of $\text{Mg}_3(\text{PO}_4)_2$ and $\text{Mg}(\text{OH})_2$ formation [2]. As a result, the best removal rates of NH_4^+ , PO_4^{3-} , and Mg^{2+} are obtained at pH = 10. However, for struvite formation, pH = 9 can be defined as the optimal treatment pH. This result is consistent with that of Booker *et al.* [27], who showed that the optimal range for struvite precipitation is in the 8.5–9.5 range.

To ensure the presence of struvite through the formed precipitates, the composition of the precipitated was characterized by X-ray diffraction, and the result is shown in Figure 8. The obtained diagram was compared to the standard ICDD database. The diagram peaks were similar to the struvite peaks (ICDD, PDF#71–2089). Similar results were also found by Taddeo *et al.* [28]. Indeed, his work recovered nutrients from raw pig manure as struvite by the addition of MgO and H_3PO_4 . Therefore, the results of this study confirmed the presence of struvite in the formed precipitates.

4. CONCLUSION

Based on the experimental results, the following conclusions for NH_4^+ and PO_4^{3-} removal from digested slaughterhouse wastewater as struvite can be drawn:

- The increase of Mg^{2+} concentration improves the removal of NH_4^+ and PO_4^{3-}
- The molar ratio $\text{Mg}^{2+} : \text{NH}_4^+ : \text{PO}_4^{3-} = 1 : 19 : 0,58$ was designated as optimal
- The pH range from pH 9 and 10 is the optimal range for struvite precipitation with optimal struvite production at pH = 9
- Above pH 10, the removal of NH_4^+ and PO_4^{3-} occurs in various forms, including struvite, hydroxyapatite, magnesium phosphate, and magnesium hydroxide
- Removal of NH_4^+ and PO_4^{3-} as struvite is possible by the addition of moderate amounts of Mg^{2+} .

5. ACKNOWLEDGMENTS

The authors express their sincere thanks to those who contributed to this study and to the reviewers and editors who helped to improve the quality of the manuscript.

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*Bibliographical Sketch



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